THERMOGRAVIMETRIC AND DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS

LOS ALAMOS
QUALITY PROGRAM

APPROVAL FOR RELEASE

D. L. BISH - PREPARER Signature on file.	DATE Date on file.
D. L. BISH - PRINCIPAL INVESTIGATOR Signature on file.	DATE Date on file.
M. J. CLEVENGER - QUALITY ASSURANCE PROJECT LEADER Signature on file.	DATE Date on file.

Los Alamos
Yucca Mountain Site Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	N/A	N/A	History of revisions not in place.
R1	N/A	N/A	History of revisions not in place. Revision 0 of this procedure was previously identified as TWS-EES-DP-107.
R2	07/11/91	3, 4, 5, & 6	Change of responsibilities. Current hardware and software used in procedure is detailed, and outdated data removed. Sections added on detection of malfunctions, safety considerations, and preparatory verification - hold points. Section added stating that curie point standards can be used to calibrate the TGAs. Added that measurements are to be documented in the controlled YMP Thermal Analysis lab logbook.
R3	04/15/97		Technical requirement revisions and minor non-substantive editorial changes.

THERMOGRAVIMETRIC AND DIFFERENTIAL SCANNING CALORIMETRY ANALYSES

1.0 PURPOSE

The purpose of this procedure is to describe the methods, procedures, and documentation used when conducting thermogravimetric (TGA) and differential scanning calorimetry (DSC) analyses.

2.0 SCOPE

This procedure applies to TGA and DSC analyses conducted for the Yucca Mountain Project.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management
LANL-EES-DP-101, Sample/Specimen Collection, Identification, and Control for Mineralogy-Petrology Studies
Polymer Laboratories, Thermal Sciences Division, PLus V, Version 5.3.2 software manual. Sales and Application Support, Amherst, MA
951 TGA Thermogravimetric Analyzer - Operator's Manual, Rev. F. (1983), DuPont Co., Wilmington, DE, pages 1-1 through 1-5 and 3-1 through 3-13.
TGA 1000/1500 Instruction Manual, PL Thermal Sciences Corporation, 300 Washington Blvd., Mundelein, IL, 60060.
910 DSC Differential Scanning Calorimeter - Operator's Manual, Rev. C. (1983) DuPont Co., Wilmington, DE, pages 1 through 9 and pages 25 through 42.

4.0 DEFINITIONS

TGA - Thermogravimetric Analyzer.

DSC - Differential Scanning Calorimeter

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure:

- The Principal Investigator (PI)
- Users of this Procedure

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, Section 6.1.6.

- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.

- When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

6.1 Principle

The purpose of the thermogravimetric analysis is to determine weight loss or gain of volatile constituents (e.g., water) from rocks and minerals during controlled heating or cooling. The purpose of differential scanning calorimetry is to measure endothermic/exothermic reactions during controlled heating of rocks and minerals. These instruments can also be operated isothermally.

6.2 Equipment and Hardware/Software

- DuPont 910 DSC

- DuPont 951 TGA

- PL Thermal Sciences TGA 1000

- PL Thermal Sciences Controllers, Power Supplies, and Computer

- PL Thermal Sciences Computer Software Package

NOTE: PL Thermal Sciences bought OMNITHERM which was the original manufacturer of the PL Thermal Sciences equipment and software.

6.2.1 Equipment Malfunctions

Equipment malfunctions should be readily apparent. The equipment either works or does not.

6.2.2 Safety Considerations

The instruments can heat samples up to 1000°C using small furnaces. Appropriate precautions shall be taken to prevent burns to the operator. Generally, the furnaces are allowed to cool off to room temperature before changing the sample.

6.2.3 Special Handling

None.

6.3 Preparatory Verification

6.3.1 Hold Points

None.

6.3.2 Calibration

6.3.2.1 Calibration of the 951 TGA balance will be performed yearly with a calibrated weight set in accordance with the 951 TGA Thermogravimetric Analyzer - Operator's Manual. A software calibration may be conducted using the Polymer Laboratories, Thermal Sciences Division, PLus V, Version 5.3.2 software manual.

6.3.2.2 Calibration of the TGA 1000 balance shall be conducted in accordance with the Polymer Laboratories, Thermal Sciences Division, PLus V, Version 5.3.2 software manual and the TGA 1000/1500 Instruction Manual.

6.3.2.3 Thermal calibration of the TGAs shall be conducted yearly using either TGA Curie point standards (traceable either to NIST or known physical constants) or a calibrated thermocouple and shall be documented in the YMP Thermal Analysis Lab logbook.

6.3.2.4 Calibration of the 910 DSC module (both enthalpy and temperature) shall be performed yearly or before use following the 910 DSC Differential Scanning Calorimeter - Operator's Manual, using NIST standards, and shall be documented in the YMP Thermal Analysis lab logbook. A software calibration may be be conducted following the Polymer Laboratories, Thermal Sciences Division, Plus V, Version 5.3.2 software manual.

6.3.2.5 Calibration stickers shall be placed on the TGA and DSC modules after calibration, and the calibration conditions shall be noted in the controlled YMP Thermal Analysis lab logbook.

6.3.2.6 Further evidence of calibration shall be accomplished in accordance with the QP-12.3.

6.3.3 Environmental Conditions

The TGA is extremely sensitive to vibration and static electricity. It should be placed on a heavy table (e.g., marble) in a location where it is not susceptible to vibrations or being "bumped" during a sample run. Various methods may be used to try to reduce any static charge (i.e., use of antistatic mats, increased humidity, etc.).

6.4 Control of Samples

Samples will be tracked, handled, shipped, and stored in accordance with DP-101.

6.5 Implementing Procedure

6.5.1 General operations of the thermal analysis system are summarized in the Polymer Laboratories, Thermal Sciences Division, PLus V, Version 5.3.2 software manual.

6.5.2 Operation of the 951 TGA module is summarized in the 951 TGA Thermogravimetric Analyzer Operator's Manual. The operator should be familiar with Sections 1 and 3 of this manual. Routine operations are described in Part 3.

6.5.3 Operation of the TGA 1000 module is summarized in the TGA 1000/1500 Instruction Manual.

6.5.4 Operation of the 910 DSC system plug-in module shall follow the 910 Differential Scanning Calorimeter-Operator's Manual. The appropriate sections of this manual regarding routine operation are Part 1, Introduction and Part 3, Operation.

6.6 Data Acquisition and Reduction

The data are analyzed using the Polymer Laboratories, Thermal Sciences Division, PLus V, Version 5.3.2 software manual. Acceptance criteria are addressed in section 8.0 of this procedure.

6.7 Potential Sources of Error and Uncertainty

Errors generally are readily apparent in the data. Potential sources of error are as follows:

- Excessive vibration or bumping the TGA during a sample run, resulting in noise spikes or large abrupt changes in signal;
- Allowing the thermocouple to contact the TGA sample pan, resulting in an uneven and unsteady background weight;
- Unstable and uneven TGA signal due to static electricity problems;
- Improper weighing of the DSC pans (e.g., handling the pans with fingers, leaving oil on them);
- Sample weighing error in DSC analysis, resulting in inaccurate energy measurements per unit mass;
- DSC analysis of a sample giving off a vapor without the use of a vapor outlet in the sample pan resulting in a large, abrupt DSC signal;

- Power failure during a run.

7.0 RECORDS

Records generated as a result of this DP are entries in laboratory notebooks or attachments to laboratory notebooks. The documentation should consist of any applicable items identified in Section 6.0 of this procedure. Laboratory notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing Center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

8.1 TGAs and DSCs are sensitive instruments, subject to a number of environmental and instrumental perturbation. Deficiencies in data are most commonly discrete shifts or noise in baseline. If significant shifts occur, the data must be assessed for usefulness and applicability to answering the questions at hand. Rejected data shall be so designated in a numbered YMP logbook and on the data plot in the binder. It is the responsibility of the analyst to determine whether the data are acceptable based on the precision needed to address his research goals.

8.2 The logbook entry in the Thermal Analysis Lab logbook for a sample shall constitute evidence that the procedure has been implemented and satisfactorily accomplished for that sample.

9.0 TRAINING

9.1 Prior to conducting work described in Section 6.0, the user requires training to this procedure.

9.2 Training to this procedure is accomplished by “read only”. Training will be documented per QP-02.7.

10.0 ATTACHMENTS

None.